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(U) FORMULATION AND EVALUATION OF INFO-635 IN DOUBLE-BASE PROPELLANTS

THOMAS J. GILDING, 1/LT, USAF

TECHNICAL REPORT AFRPL-TR-65-219

JANUARY 1966

AIR FORCE ROCKET PROPULSION LABORATORY
RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE
EDWARDS, CALIFORNIA

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FOREWORD

(U) This report describes the development of techniques for formulating and evaluating the high energy fluorine-oxygen oxidizer, INFO-635, in a double-base system. The work was carried out under Project 314804203, Formulation and Evaluation of Solid Propellants, by the Evaluation and Test Branch, Propellant Division of the Air Force Rocket Propulsion Laboratory from July 1964 to July 1965.

This report has been reviewed and approved.

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ABSTRACT

(C) The NF solid oxidizer INFO-635, (2-tris (difluoramino) methoxy) ethyl ammonium perchlorate, was successfully formulated into two double-base binder systems. Parameters affecting the impact sensitivity of the 635 propellant were investigated. Strand burnings were also conducted using both binder systems. The highest solid loading of INFO-635 for strand burning tests was 25%. INFO-635 was added to a non-NF solid propellant to determine the feasibility of using it as a burn-rate modifier.

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(U) FORMULATION AND EVALUATION OF INFO-635 IN DOUBLE-BASE PROPELLANTS

I. INTRODUCTION.

(C) During the past five years a large emphasis has been placed on the synthesizing of new high-energy compounds for rocket propellant applications. Within this effort is an area concerned with compounds containing the diflour-amino group (NF_2). INFO-635 was one of the first promising solid NF oxidizers to become available for propellant evaluation from this effort.

(C) The Minnesota Mining and Manufacturing Company (3M) was the first to synthesize this compound by adding ethanolamine perchlorate to perfluoroguanidine, and flourinating the resulting adduct. INFO-635 is a white, solid ionic salt, represented by the chemical formula, $(NF_2)_3COCH_2$-CH_2NH_3 CLO_4.

(U) The Air Force Rocket Propulsion Laboratory soon followed 3M in the production of INFO-635 (1). The main supply of INFO-635 for the investigation described in this report was prepared in-house.

(C) The objective of this program was to incorporate INFO-635 into a double-base propellant system having a theoretical Isp of 280 seconds. A secondary objective was to determine the feasibility of using INFO-635 as a burn-rate modifier in non-NF solid propellants.

II. (U) FORMULATION OF INFO-635.

A. Compatibility Tests.

Compatibility tests indicated that the INFO-635 first received was not compatible with the standard double-base plasticizers, trimethylolethane trinitrate (TMETN), diethyleneglycoldinitrate (DEGDN), triethyleneglycoldinitrate (TEGDN), adiponitrile, and succinonitrile, the compatibility determinations were conducted for 2-week periods at 40°C.

The addition of a small amount of NaF improved the compatibility between INFO-635 and the nitrate esters. There was no evidence of incompatibility after the testing period. With the nitrile plasticizers, NaF only slightly increased the induction time before a reaction became evident.

LiF accelerated, rather than inhibited, the reaction between INFO-635 and the nitriles.

A new purification procedure was developed for INFO-635 which greatly improved its quality. This procedure is similar to the purification procedure introduced by duPont (2) except that nitromethane is the extracting solvent instead of ethylacetate (Figure 1).

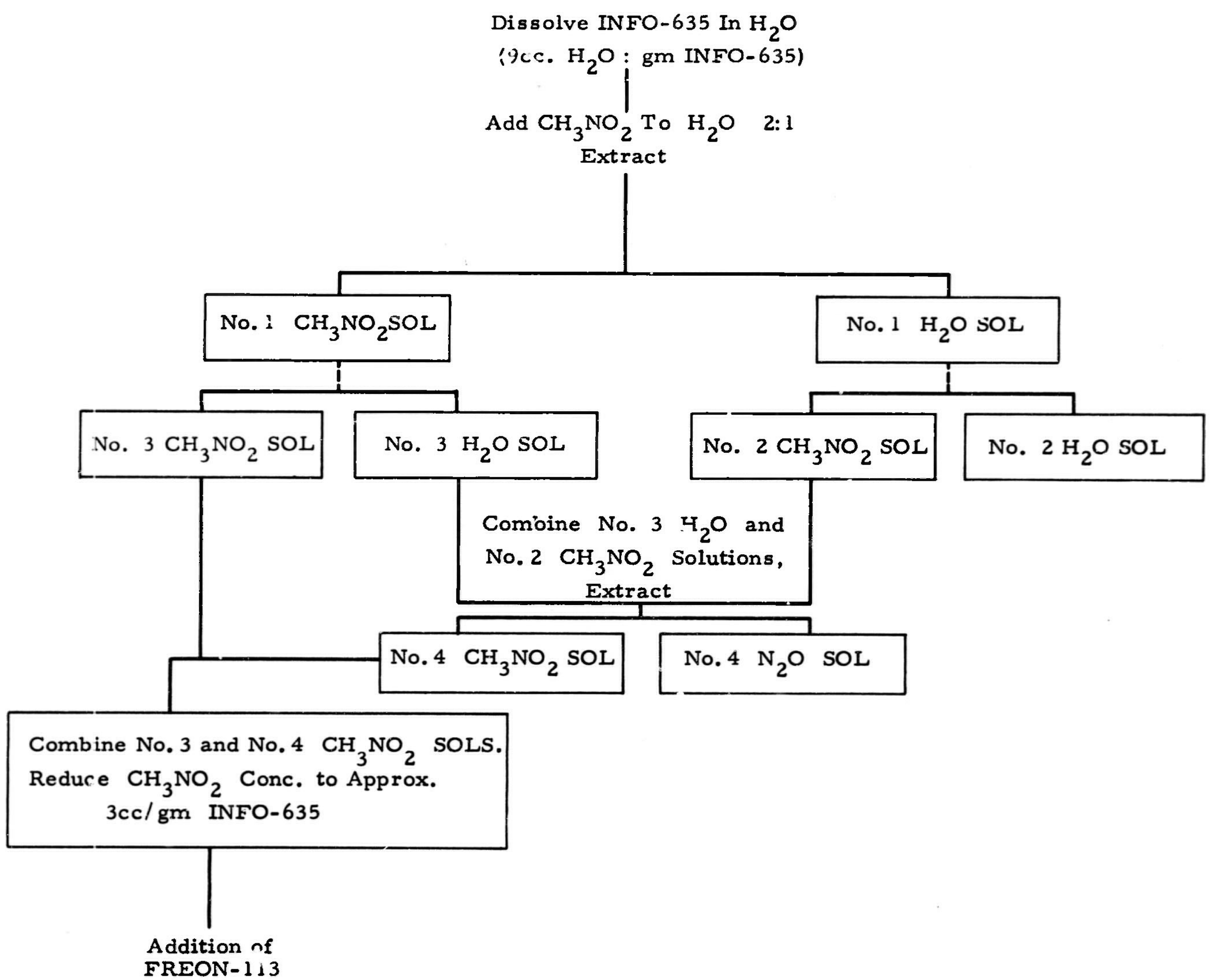
The compatibility results with the purified INFO-635 showed a similarity to those in which NaF was added. There were some tests between INFO-635 and the nitriles which had no visible reactions after the 2-week period. However, this occurred only when the sample of INFO-635 was of extremely high purity.

Since the successful formulation with nitrile plasticizers was so dependent on the purity of the INFO-635, that approach was not continued. The evaluation was continued using only the nitrate esters.

The improved INFO-635 was satisfactorily formulated into two binder systems:

I		II	
TMETN	61%	TEGDN	70%
DEGDN	13%	Nitrocellulose	30%
Nitrocellulose	26%		

Both systems cured within 24 hours at 40°C.



(U) Figure 1. Extraction Procedure for INFO-635 Purification

Compatibility tests and early attempts to cure 635 double-base propellants revealed that 45°C was the maximum safe curing temperature.[1] A small amount of 635 propellant, using binder system II, was placed in an oven at 60°C and detonated after approximately 18 hrs.

To decrease the possibility of detonations while curing 635 propellant, the decision was made to cure only at temperatures below 45°C. There have been no detonations while curing below this temperature. Propellant samples have remained at 40°C for 72 hours without any sign of decomposition. Samples of 635 propellant have also been stored at room temperature (25°C) for 1 year without any visible evidence of decomposition.

B. Mixing Preparation and Procedure.

The initial step in preparing INFO-635 for mixing is purification. INFO-635 must be purified before mixing to ensure acceptable quality for formulation.

Few INFO-635 samples have been formulated as received. Some samples did cure satisfactorily, while others exhibited localized decolorization. This suggested the presence of reactive impurities in some of the samples received.

After the purification of INFO-635 in nitromethane and water, the nitromethane concentration is reduced in the INFO-635 - nitromethane solution to approximately 3 cc per gram of INFO-635. Freon-113 is then added to the solution at the ratio of 6/1, Freon-113/nitromethane. At this point, the nitromethane and Freon-113 become miscible. Methylene chloride is then added to the solution to obtain a more complete precipitation of INFO-635.

INFO-635 prepared in this manner is precipitated and freon-washed (3) in one step.

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[1] Propellant formulations containing INFO-635 will be referred to in this report as 635 propellant.

In order to eliminate the flakes of INFO-635 that were evident in the early propellant, the oxidizer was broken up and passed through an 80-mesh sieve. The oxidizer was broken up by brushing a small amount (approximately 100 mg) at one time over filter paper.

INFO-635 which was precipitated in the presence of Freon-113 was much easier to get into a powdery form. (Figure 2 shows the sieve and the apparatus used in breaking up the oxidizer.)

The laboratory mixing of 635 propellant was done remotely under vacuum on a 6g scale (Figures 3 and 4).

III. (U) EVALUATION OF 635 PROPELLANT.

A. Impact Sensitivity.

Impact sensitivity of the 635 propellant was determined on the Olin Mathieson drop-weight tester, which had been modified for solid samples. The thickness of the propellant slices was 0.01 inch.

The concentration of INFO-635 in all the propellant samples for impact sensitivity determinations was held constant at 20% so a standard propellant would be used throughout the testing.

The impact sensitivity[2] of the first 635 propellant (non-freon-washed, flaky INFO-635) was determined to be 3kg-cm (RH-P-112/ 12 kg-cm).[3] This is the same impact sensitivity as neat, non-freon, washed INFO-635.

The impact sensitivity of the propellant containing freon-washed, flaky INFO-635 was 9 kg-cm. This is the same as neat, freon-washed INFO-635.

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[2] All impact readings for 635 propellant are given as the point of 0% fire.

[3] Rohm & Haas Standard Plastisol Propellant.

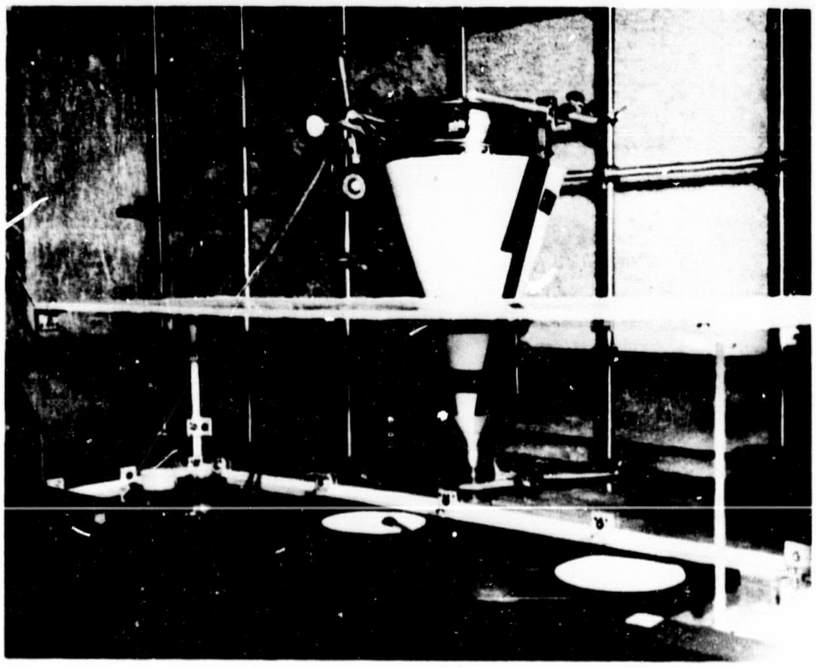

Figure 2. Plexiglass Grinding Box and Sieve (80-Mesh)

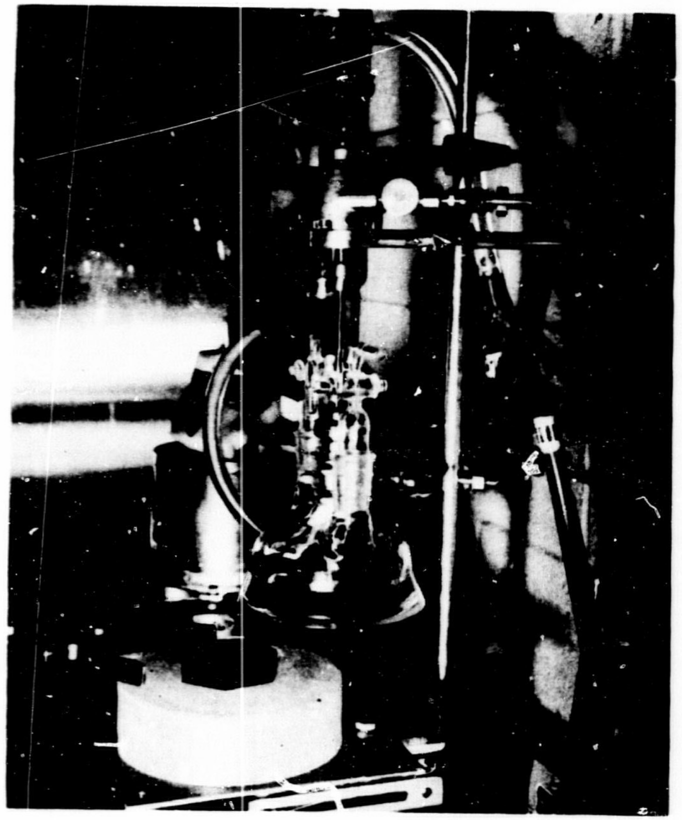

Figure 3. Remote Laboratory Mixer, Open

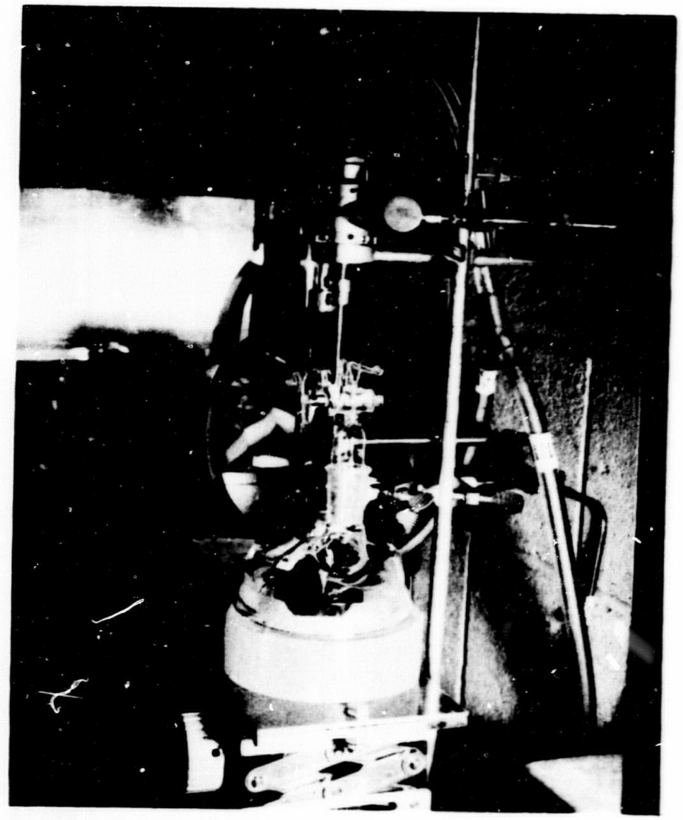

Figure 4. Remote Laboratory Mixer, Closed

It became apparent that in order to further improve the impact sensitivity of the propellant, the flakes would have to be eliminated. This was verified by incorporating freon-washed INFO-635 with a particle size no larger than 177 microns. The impact sensitivity of this propellant was determined to be 18 kg-cm.

As the concentration of INFO-635 increases, the impact sensitivity will increase. However, these preliminary tests indicate that no matter what the concentration, the propellant will be less sensitive toward impact, if the INFO-635 particles are small enough so that the binder can cushion each individual particle. If there is any INFO-635 directly exposed to the impact, the impact sensitivity of the propellant will approach that of INFO-635.

B. Strand Burning.

The strand-burning apparatus used was the Atlantic Research Corporation Model 202. The dimensions of the strands were 1/8 x 1/8 x 2 in., with a time length of $1\frac{1}{4}$ in. Figures 5 and 6 show the mold used for 635 propellant and how the propellant was removed.

The concentration of INFO-635 in the strands subjected to burn-rate determinations did not exceed 25%. Detonations were encountered in almost every attempt at this and lower levels of INFO-635 concentration.

The first attempts to burn 635 propellant were with strands containing 25% INFO-635. These strands were ignited on the strand holder in the open atmosphere and burned smoothly. When strands from this same batch were placed in the combustion bomb of the strand burner, still at atmospheric pressure but under a nitrogen atmosphere, they failed to ignite. These strands would only ignite above pressure of 150 p.s.i. When they did ignite, however, they detonated. To obtain strands that would successfully burn at desired pressures up to 1500 p.s.i., the concentration of INFO-635 was lowered to 10%. These strands would not ignite below pressures of 200 p.s.i., but above this pressure they would detonate upon ignition.

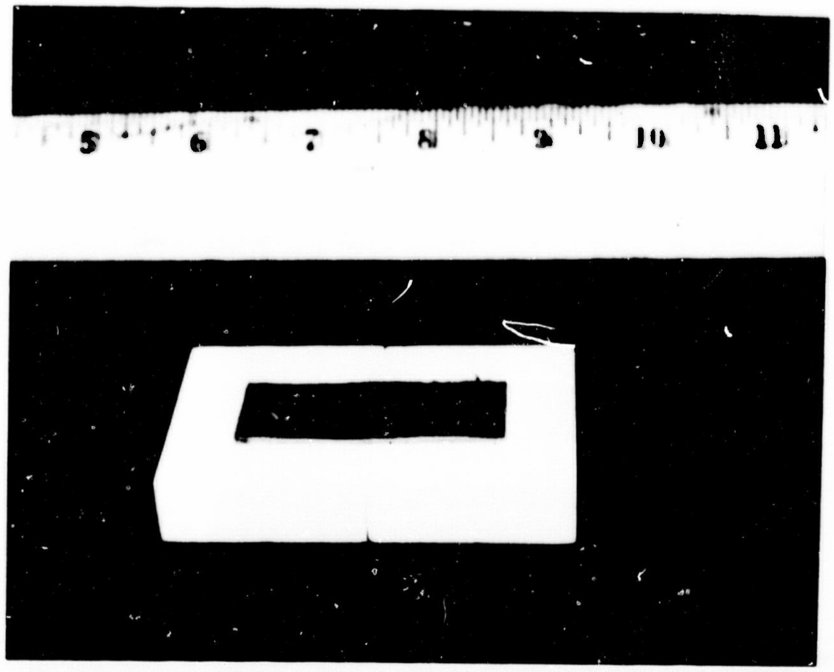

Figure 5. Remote Propellant Mold, Closed

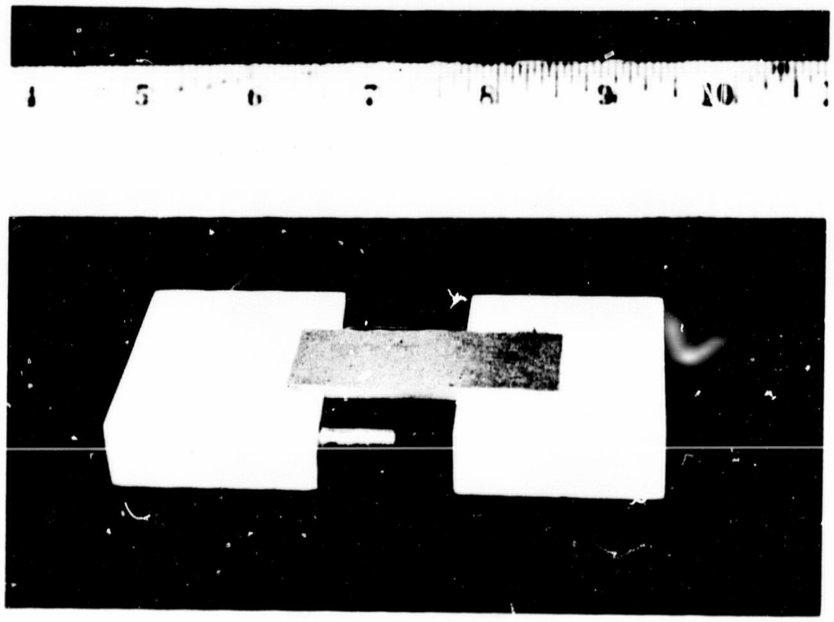

Figure 6. Remote Propellant Mold, Open

Strands containing 5% aluminum and 25% INFO-635 were also burned. The results from six of these tests are listed in Table I.

TABLE I

STRAND-BURNING RESULTS OF ALUMINIZED INFO-635 STRANDS

Test No.	Pressure p.s.i.	Length Burned (inches)	Duration (sec.)	Comment
1	200	$1\frac{1}{4}$	0.55	Detonated
2	200	$1\frac{1}{4}$	1.83	Detonated
3	200	$1\frac{1}{4}$	0.00	Detonated
4	200	$1\frac{1}{4}$	0.80	Detonated
5	200	$1\frac{1}{4}$	0.06	Detonated
6	200	$1\frac{1}{4}$	0.00	Detonated

All strands did detonate. However, as can be seen, the detonations occurred at different intervals after ignition. Even strands 3 and 6 could have detonated sometime after ignition because of the 3/4-in. length between the igniting wire and the first fuse wire.

No further investigations in strand burning were conducted. The main reason for the termination was that INFO-635 was not considered a promising candidate for propellant use. This was because of its unstable combustion behavior, and the continued high impact sensitivity and thermal instability problems.

C. Burn-rate modifier.

A preliminary investigation to determine the feasibility of using INFO-635 as a burn-rate modifier in solid propellants was conducted. A standard double-base formulation, shown below, was chosen as the baseline propellant, 635-20.

Ingredient	Wt. %
TEGDN	9
TMETN	29
Nitrocellulose	12
AP	30
AL	20

The burn rate of the baseline propellant was determined, at 500 p. s. i., to be 0.59 in/ sec.

INFO-635 was added to the baseline formulation at a concentration of 5%, reducing AP from 30% to 25%. In the first attempt, 635-20-1, the propellant cured without any compatibility problems. The cured propellant did appear to be slightly porous. This was probably caused by the INFO-635 dissolving into the TEGDN after the propellant had begun to set. In the next attempt, 635-20-2, INFO-635 was given ample time to dissolve in the plasticizer. There were no voids evident in the cured propellant. Despite the porosity of 635-20-1, four strands were burned to get some idea of the consistency in the combustion behavior. Table II shows the results of both 635-20-1 and 635-20-2.

After observing the results of 635-20-2, the increase in burn rate of 635-20-1 can only be attributed to the porosity of the strands.

These preliminary results indicate that 5% INFO-635 is insufficient to affect an increased burn rate. If the concentration of INFO-635 is increased (over 10%), detonations can be expected.

IV. (U) THEORETICAL CALCULATIONS.

Table III lists the theoretical Isp of practical 635 formulations.

The addition of up to 10% of aluminum had an increasing effect on the specific impulse. Above this concentration the specific impulse began to lower.

TABLE II

STRAND-BURNING RESULTS IN DETERMINATION OF INFO-635 AS A BURN-RATE MODIFIER

Modified Baseline Formulation	Test No.	Pressure (p.s.i.)	Length Burned (in.)	Duration (sec.)	Average Burn Rate (in./sec.)
635-20-1	1	500	$1\frac{1}{4}$	1.58	0.80[a]
	2	500	$1\frac{1}{4}$	1.60	
	3	500	$1\frac{1}{4}$	1.50	
	4	500	$1\frac{1}{4}$	1.57	
635-20-2	1	500	$1\frac{1}{4}$	2.30	0.58[b]
	2	500	$1\frac{1}{4}$	2.00	
	3	500	$1\frac{1}{4}$	2.20	
	4	500	$1\frac{1}{4}$	2.13	

[a] Average of four tests, Nos. 1, 2, 3, 4, propellant formulation 635-20-1.

[b] Average of four tests, Nos. 1, 2, 3, 4, propellant formulation 635-20-2.

TABLE III

(C) THEORETICAL PERFORMANCE OF DOUBLE-BASE FORMULATIONS CONTAINING INFO-635 WITH ALUMINUM
ΔH_F(INFO-635) = -115 Kcal/mole

NC	COMPOSITION TMETN	DEGDN	(WT.%) INFO-635	AL	SPECIFIC IMPULSE (LB-SEC/LB)
15.5	36.0	8.5	40	--	262.1
14.5	33.0	7.5	40	5	268.8
13.0	30.0	7.0	40	10	273.1
12.0	27.0	6.0	40	15	273.6
10.5	24.0	5.5	40	20	270.7
13.0	30.0	7.0	50	--	267.0
12.0	27.0	6.0	50	5	272.4
10.5	24.0	5.5	50	10	275.5
9.3	21.0	4.7	50	15	275.0
8.0	18.0	4.0	50	20	271.4
10.5	24.0	5.5	60	--	271.6
9.5	21.0	4.5	60	5	275.6
8.0	18.0	4.0	60	10	277.4
6.6	15.0	3.4	60	15	276.2
5.3	12.0	2.7	60	20	272.2

Figure 7 shows the effect of the concentration level of aluminum on the specific impulse of an INFO-635 double-base formulation.

The substitution of beryllium metal for aluminum metal provides a substantial increase in the specific impulse, as shown in Figure 8.

The use of beryllium in this program was not considered because of the toxicity problems in conducting test operations.

V. (U) FUTURE PLANS AND COMMENTS.

The information obtained from this evaluation does not justify the use of INFO-635 in future solid propellants. For this reason, the in-house evaluation of INFO-635 has been terminated. The undesirable combustion and sensitivity properties of INFO-635 have eliminated it from consideration as a future solid propellant ingredient (4).

Even though it is quite certain that INFO-635 will never obtain the status of a standard propellant ingredient, the investigation conducted was worth the expended effort. INFO-635 can be considered a "model compound" for future NF compounds. The information obtained from this evaluation can be applied to future NF evaluations.

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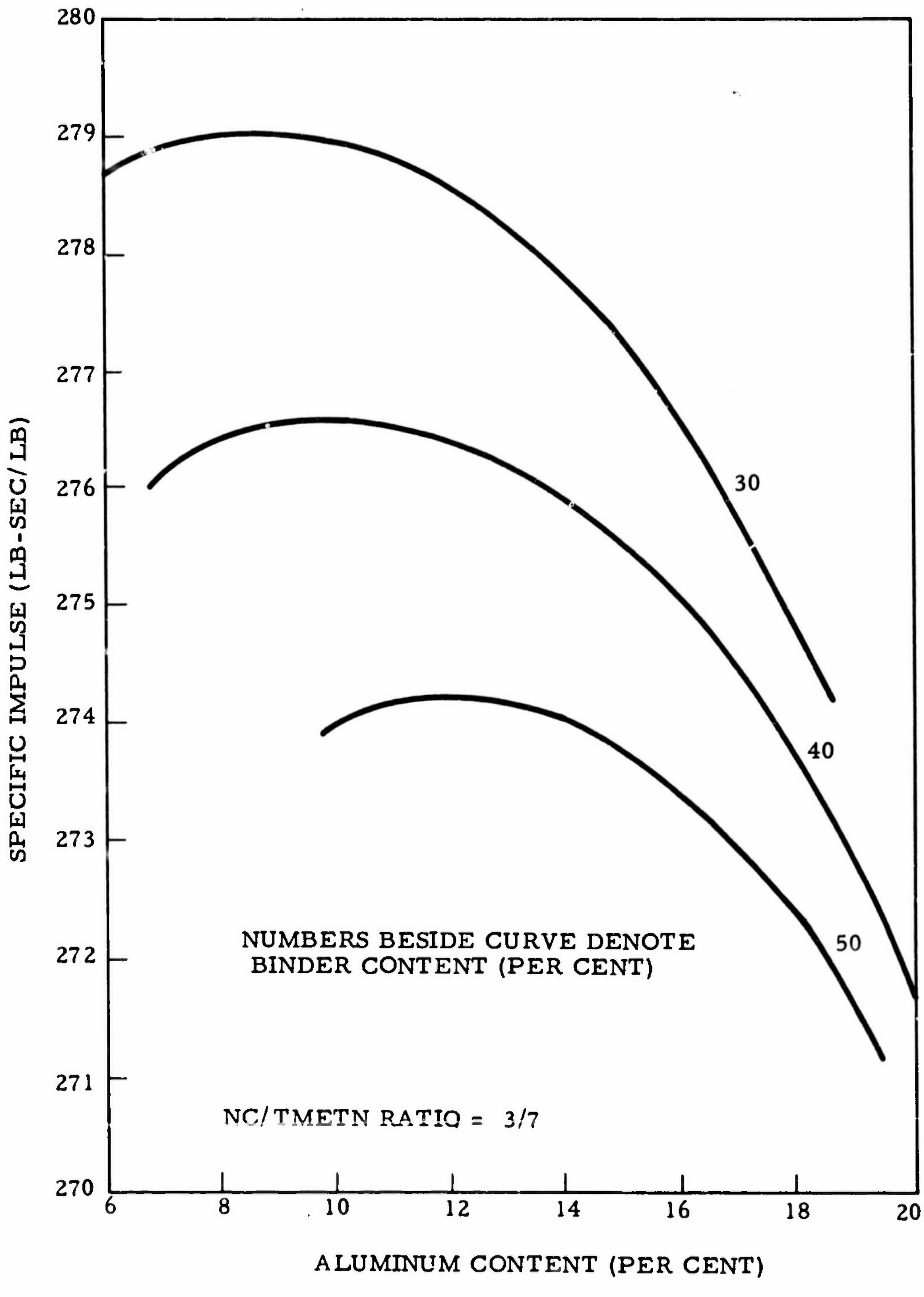

Figure 7. Theoretical Specific Impulse of Formulations Consisting of NC, TMETN, Aluminum, and INFO-635 (Reference 5)

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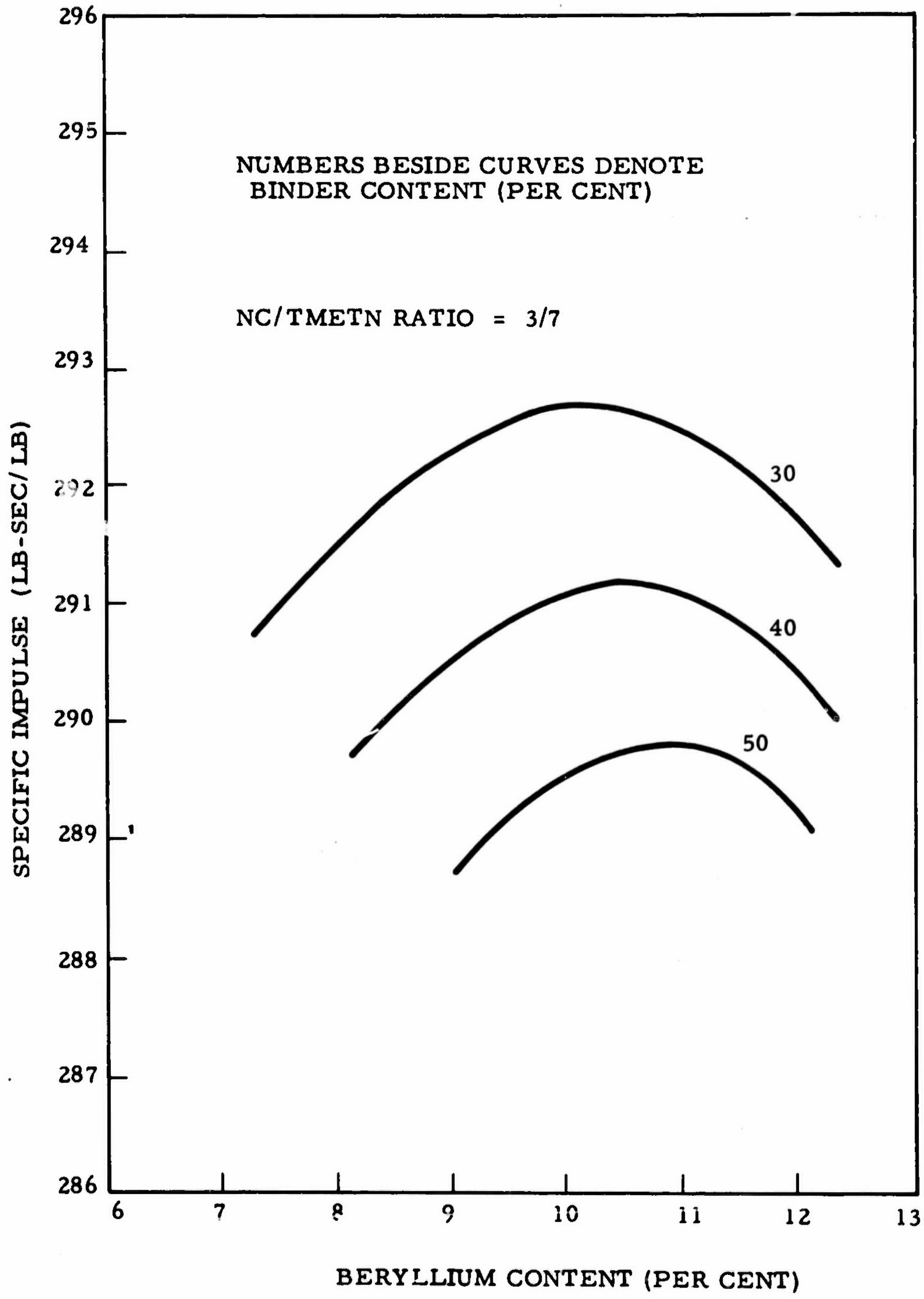

Figure 8. Theoretical Specific Impulse of Formulations Consisting of NC, TMETN, Beryllium, and INFO-635 (Reference 5)

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